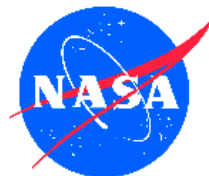


# **Combined Thermomechanical and Environmental Durability of Environmental Barrier Coating Systems on SiC/SiC Ceramic Matrix Composites**

Dongming Zhu, Bryan Harder and Ramakrishna Bhatt

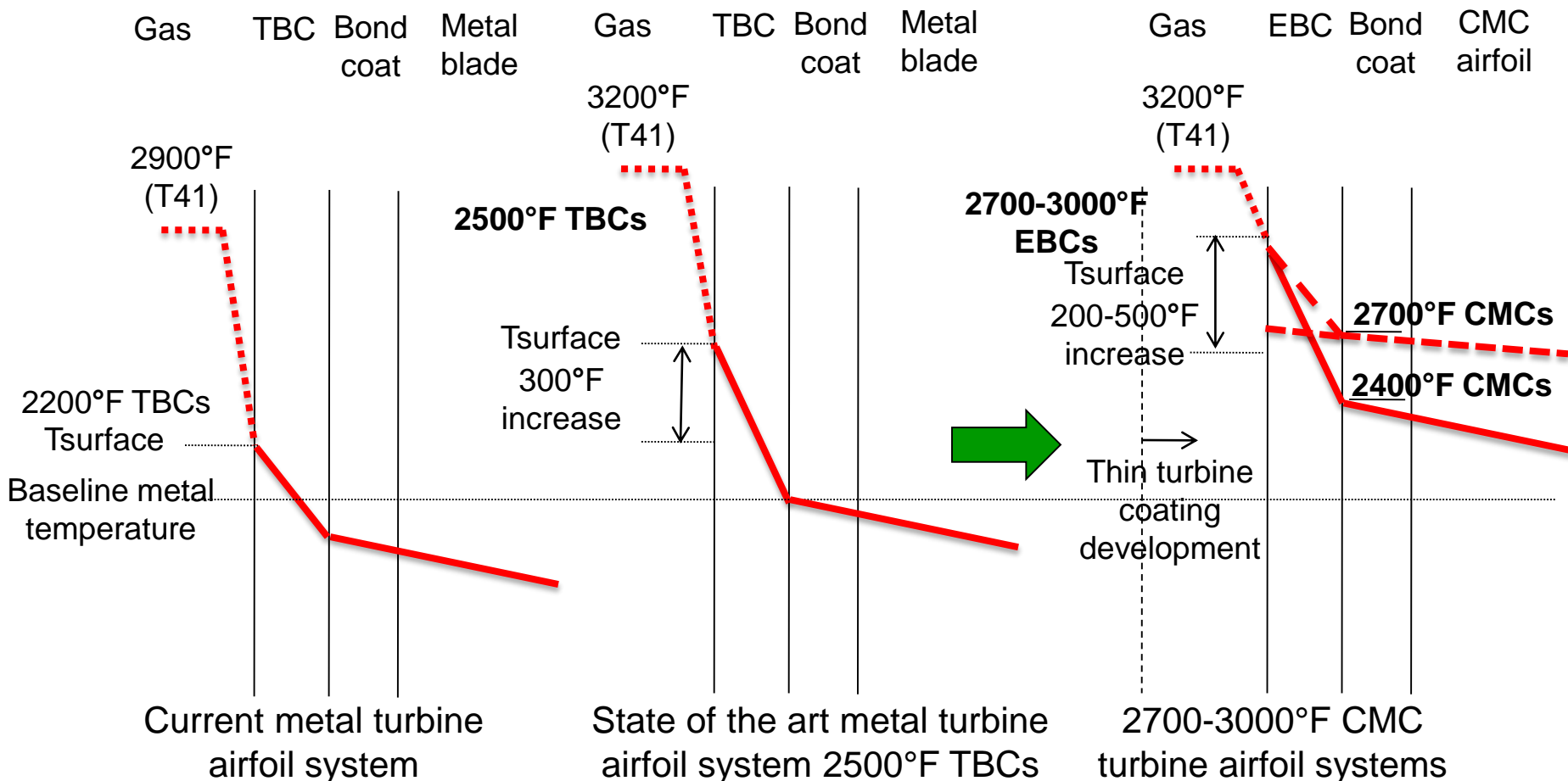
Materials and Structures Division  
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Cleveland, Ohio 44135, USA



**9th International Conference on High Temperature Ceramic Matrix Composites (HTCMC-9)**  
**Toronto, Canada**  
**June 26-July 1, 2016**

# NASA Turbine Environmental Barrier Coatings for CMC-EBC Systems

- Emphasize temperature capability, performance and durability for next generation turbine engine systems
- Increase Technology Readiness Levels for component system demonstrations





## **Environmental Barrier Coating and SiC/SiC System Development: Testing Challenges**

- High Temperatures: 2700 to 3000°F (1500-1650°C) along with higher interface temperatures
- Exposure to water vapor and combustion products
- High Cyclic Stresses: thermal and mechanical, creep-fatigue effect
- Combined Interactions, in-plane and through-thickness gradients
- High Velocity Gases: Mach 1 and 2
- High Pressures: ~ up to 40 to 50 atmospheres
- Long term durability: 20,000 hr design life



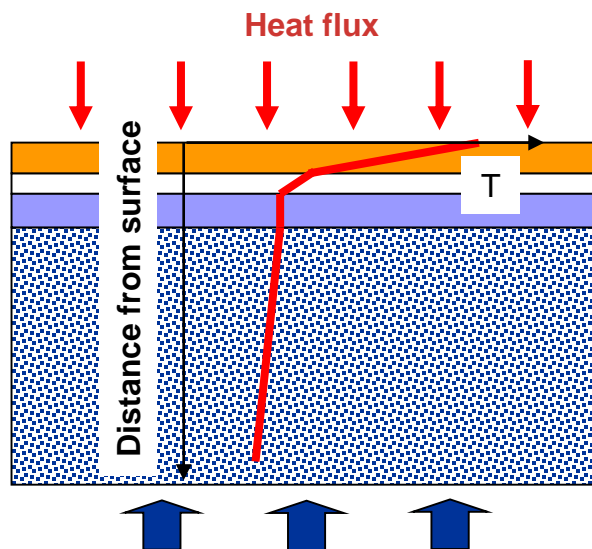
## Outline

- **Advanced testing approaches for SiC/SiC and ceramic coating development: laser high heat flux based testing approaches**
  - NASA CO<sub>2</sub> laser rig development
  - Thermal conductivity
  - Cyclic durability and monitoring degradations of EBCs and CMCs
- **Laser high heat flux and mechanical tests**
  - Combined high heat flux - mechanical tests
  - High heat flux biaxial creep/fatigue test rigs
  - Sub-element testing
- **Summary and future directions**

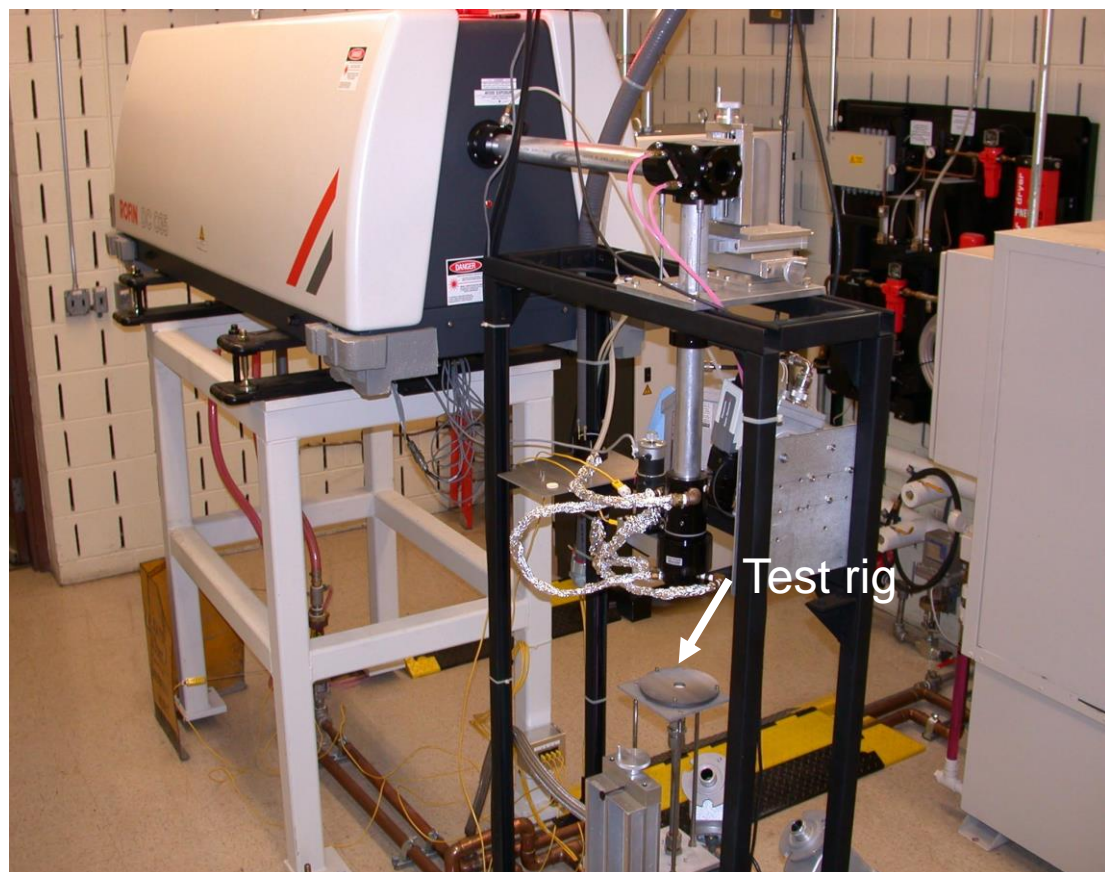
# High Power CO<sub>2</sub> Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development

- Developed in 1990's, the rig achieved turbine level high-heat-fluxes (315 W/cm<sup>2</sup>) for turbine thermal barrier coating testing
- Crucial for advanced EBC-CMC developments

Turbine: 450°F across 100 microns  
 Combustor: 1250°F across 400 microns



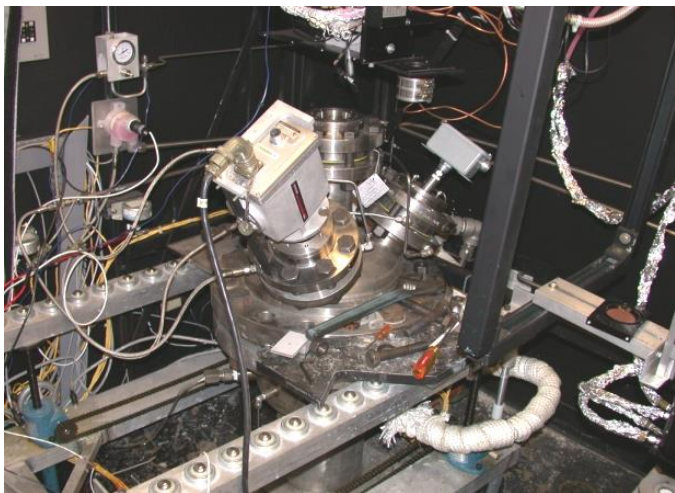
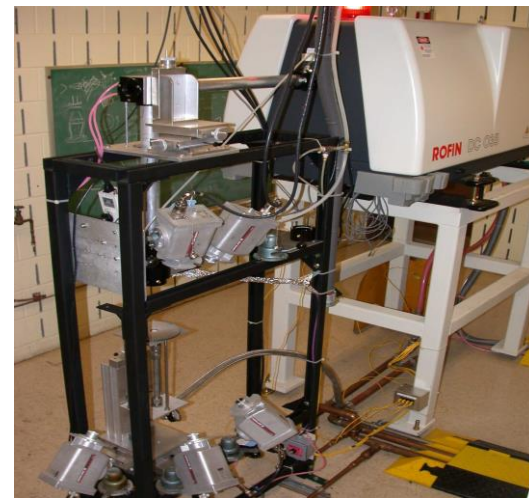
Achieved heat transfer coefficient 0.3 W/cm<sup>2</sup>-K



# High Power CO<sub>2</sub> Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development

## - Continued

- NASA high power CO<sub>2</sub> laser rig systems
- Various test rigs developed
- 7.9 micron single wavelength and 1 micron two color wavelength pyrometers for temperature measurements
- Thermography system for temperature distribution measurements
- Capable of programmable test mission cycles
- Capable of mechanical load cycles under high heat flux
- Environment test conditions (e.g., steam and vacuum)



Some temperature thermal gradient cycles



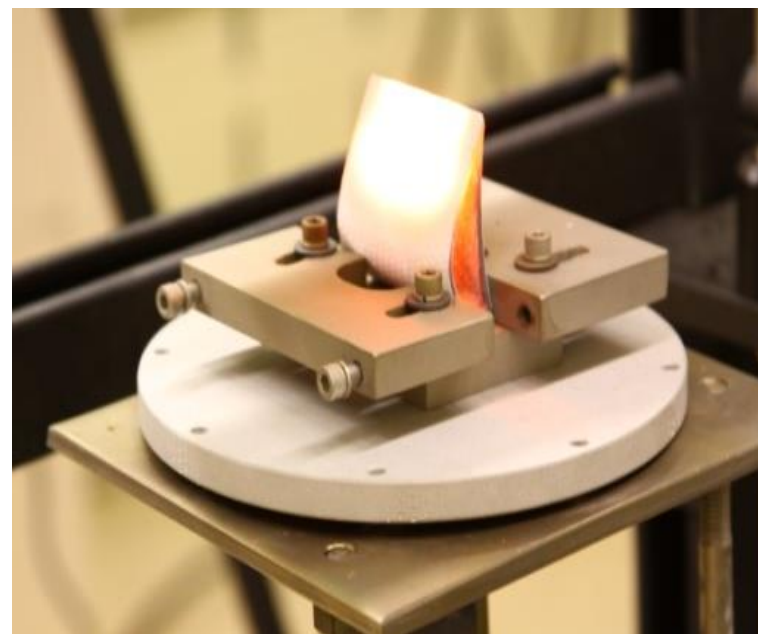
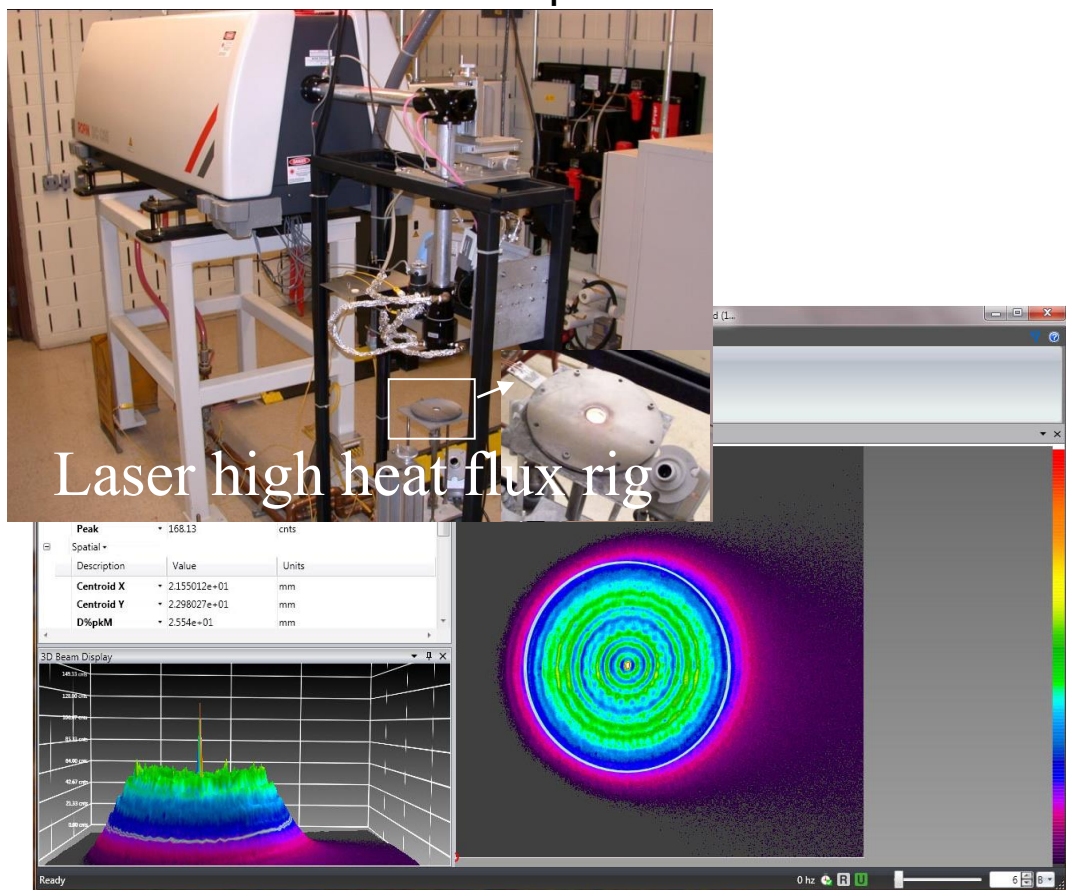
High heat flux combustor rig



# High Power CO<sub>2</sub> Laser Based High Heat Flux Testing for SiC/SiC and Environmental Barrier Coatings Development

## – Continued

- Controlled beam profiles, beam size and power density were major emphases, by using rotating ZnSe integrating lens with various focus lengths
- Uniform distribution up to 2-3" diameter beam size for various testing

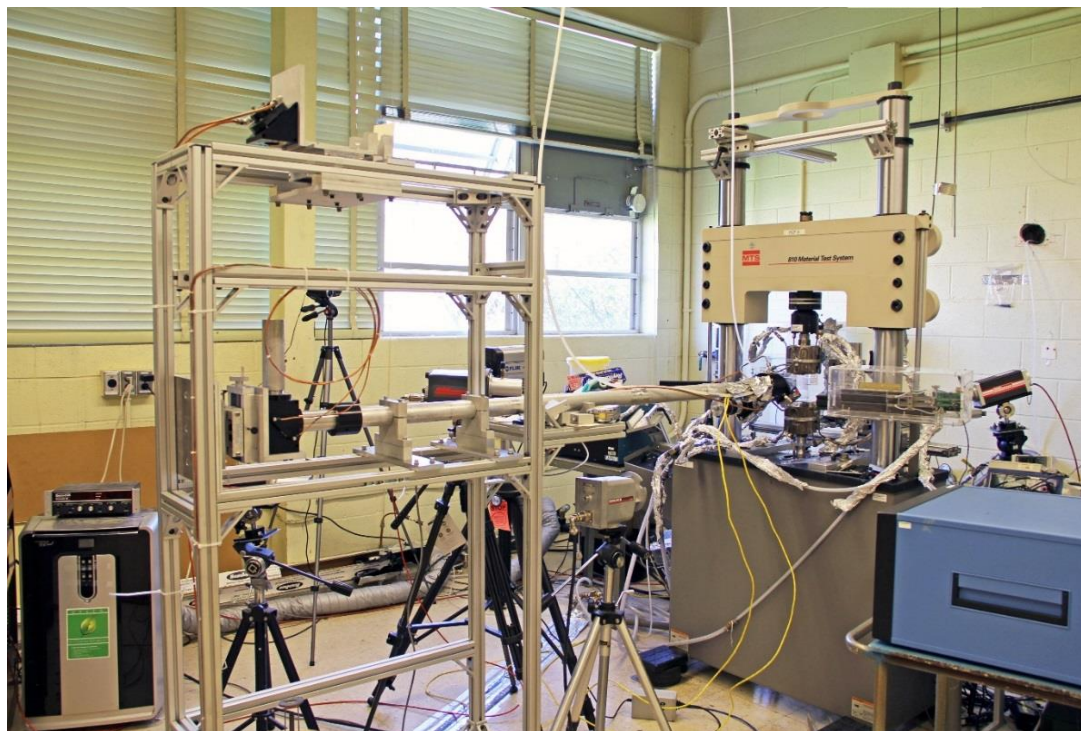


2" beam size subelement tests

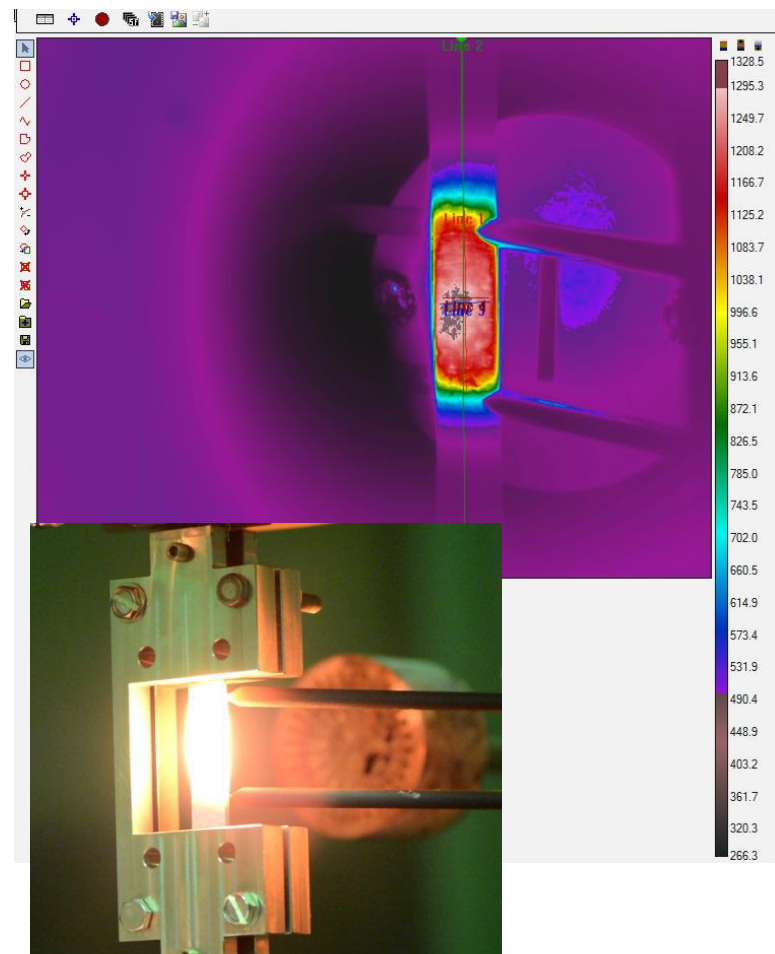
Example of 1" diameter disc specimen tests and beam profile

# High Power CO<sub>2</sub> Laser Based High Heat Flux Fatigue Test Rig

- Laser creep and fatigue testing capable of full tension and compression loading
- Uniform distribution up to 2-3" diameter beam size for various testing, depending on the heat flux requirements



Laser heat flux Thermal HCF/LCF Rig – Overall View

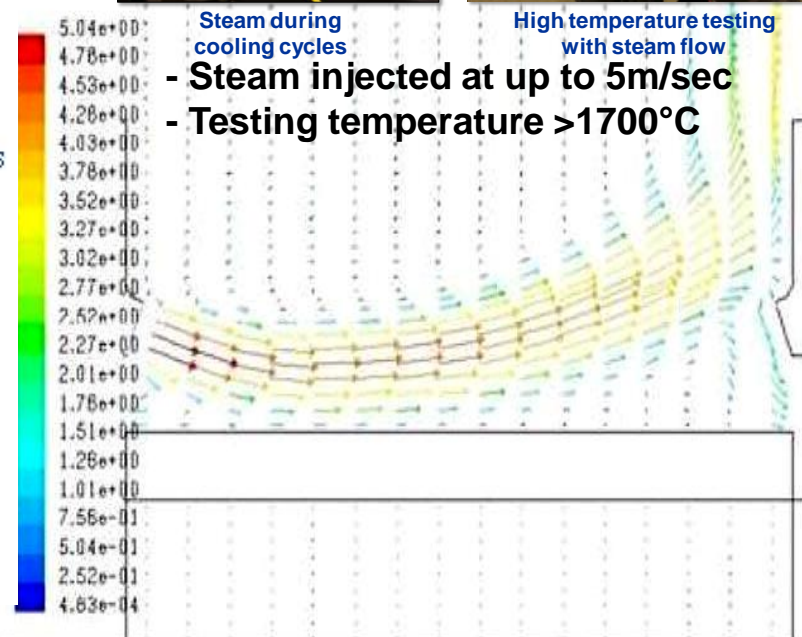
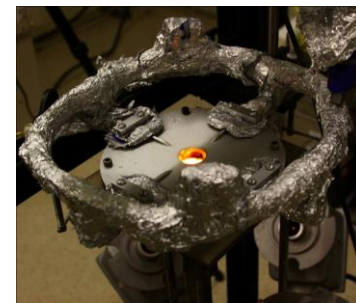
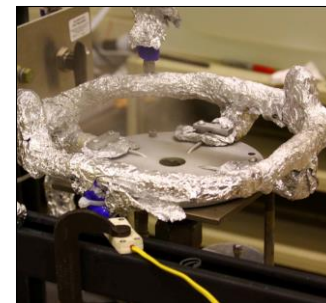


Specimen under testing in tensile-compression fatigue rig



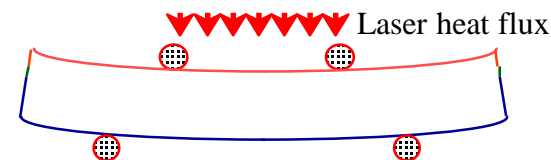
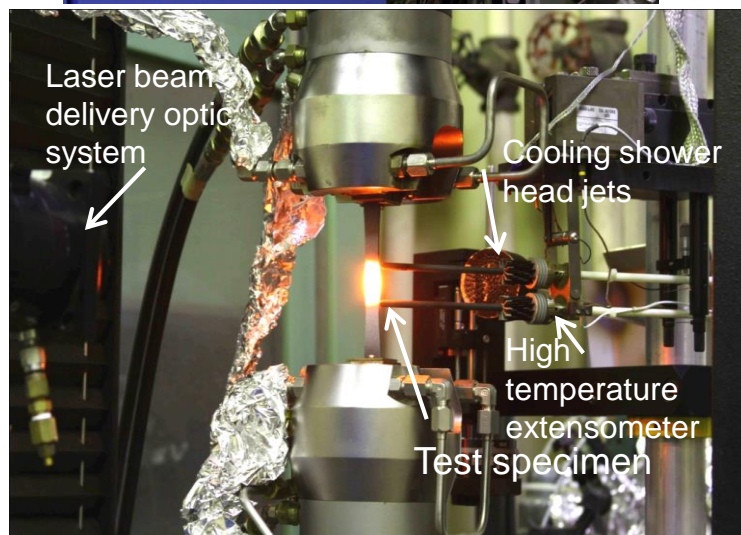
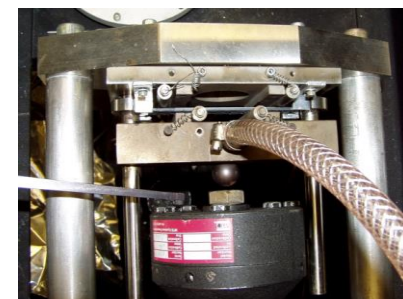
## High Heat Flux Rig Testing with Water vapor Steam Chamber – Established in Early 2000

- High temperature and high-heat-flux testing capabilities
- “Micro-steam environment” allowing high water vapor pressure, relatively high velocity under very high temperature condition
- Used for 3000°F EBC-CMC developments



# High Heat Flux Thermomechanical Testing for EBC Development

- High heat flux and combined thermal-mechanical loading capabilities established to allow SiC/SiC system performance data to be obtained under simulated operating conditions
- A 1000 Hz high heat flux HCF testing rig is being established this year



High heat flux flexural TMF testing: HCF, LCF, interlaminar and biaxial strengths

High heat flux tensile TMF and rupture testing

# Thermal Conductivity Measurement by a Laser High-Heat-Flux Approach

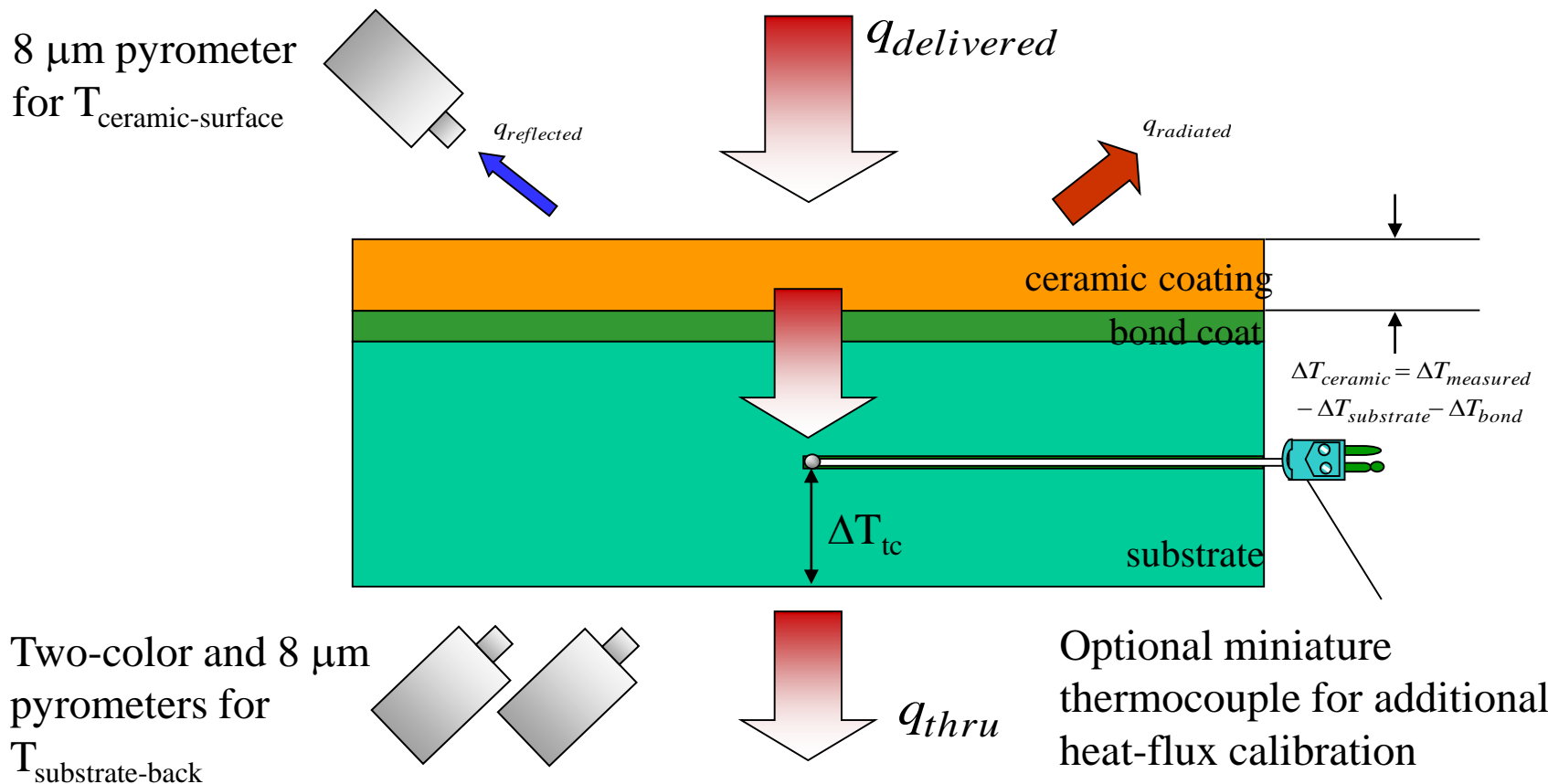
$$k_{ceramic}(t) = q_{thru} \cdot l_{ceramic} / \Delta T_{ceramic}(t)$$

$$q_{thru} = q_{delivered} - q_{reflected} - q_{radiated}$$

and

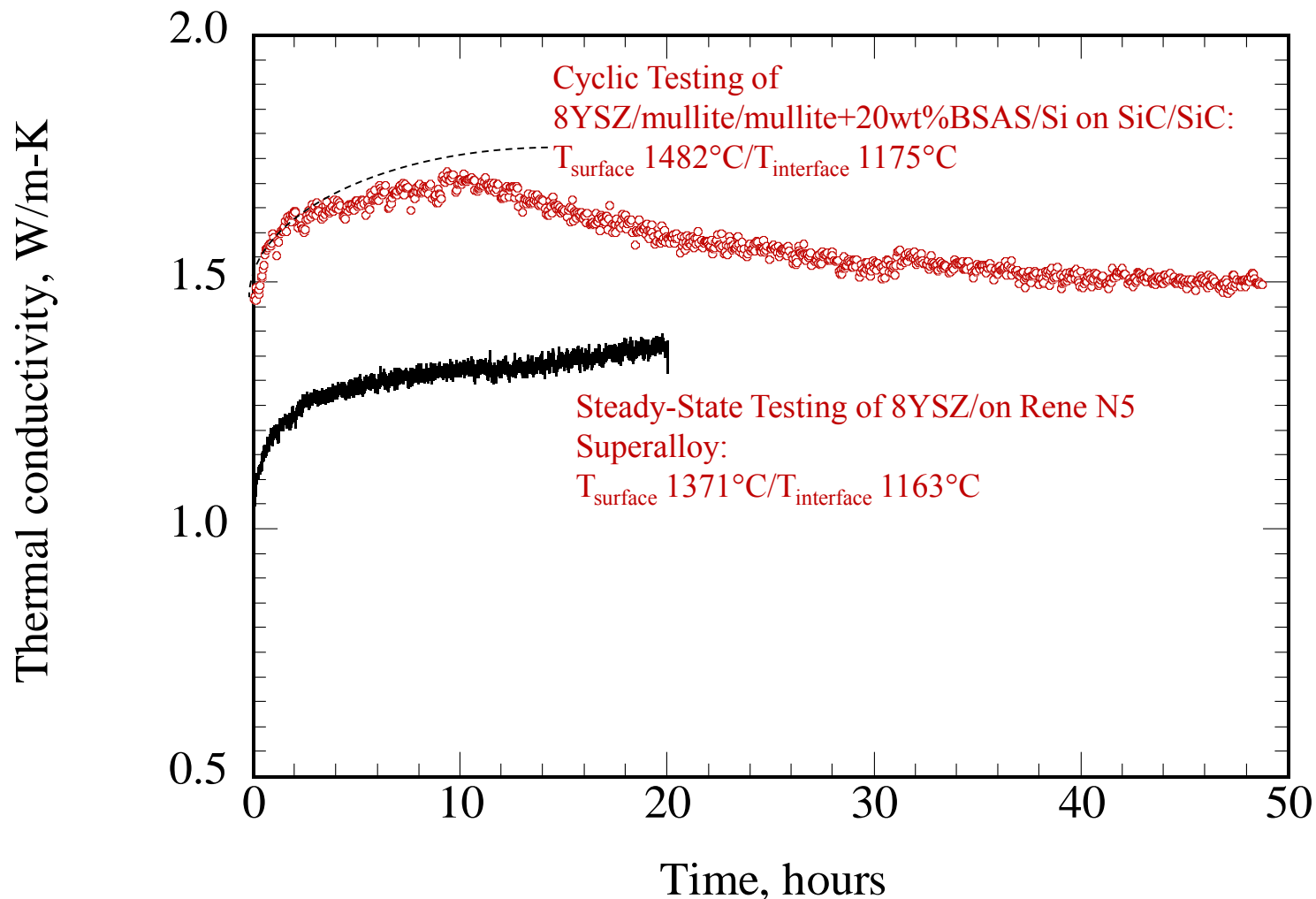
$$\Delta T_{ceramic}(t) = T_{ceramic-surface} - T_{metal-back} - \int_0^{l_{bond}} \frac{q_{thru} \cdot dl}{k_{bond}(T)} - \int_0^{l_{substrate}} \frac{q_{thru} \cdot dl}{k_{substrate}(T)}$$

Where



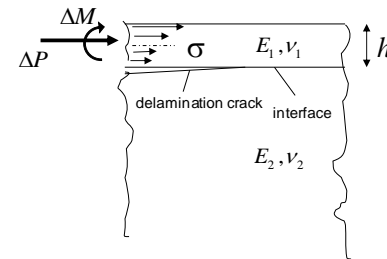
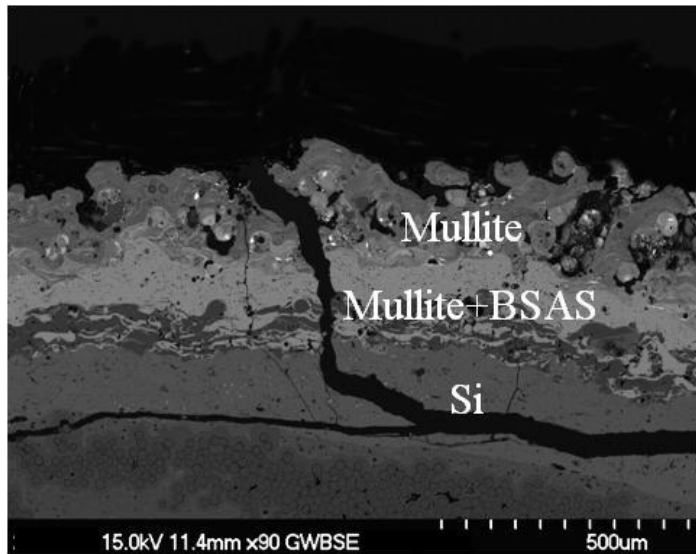
## Thermal Gradient Cyclic Behavior of a Thermal Environmental Barrier Coating System

- Sintering and delamination of coatings reflected by the apparent thermal conductivity changes



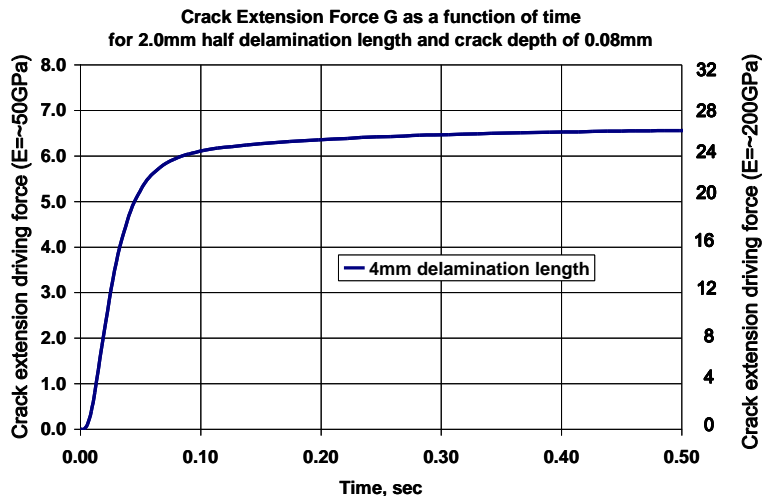


# Environmental Barrier Coating and High Heat Flux Induced Delaminations

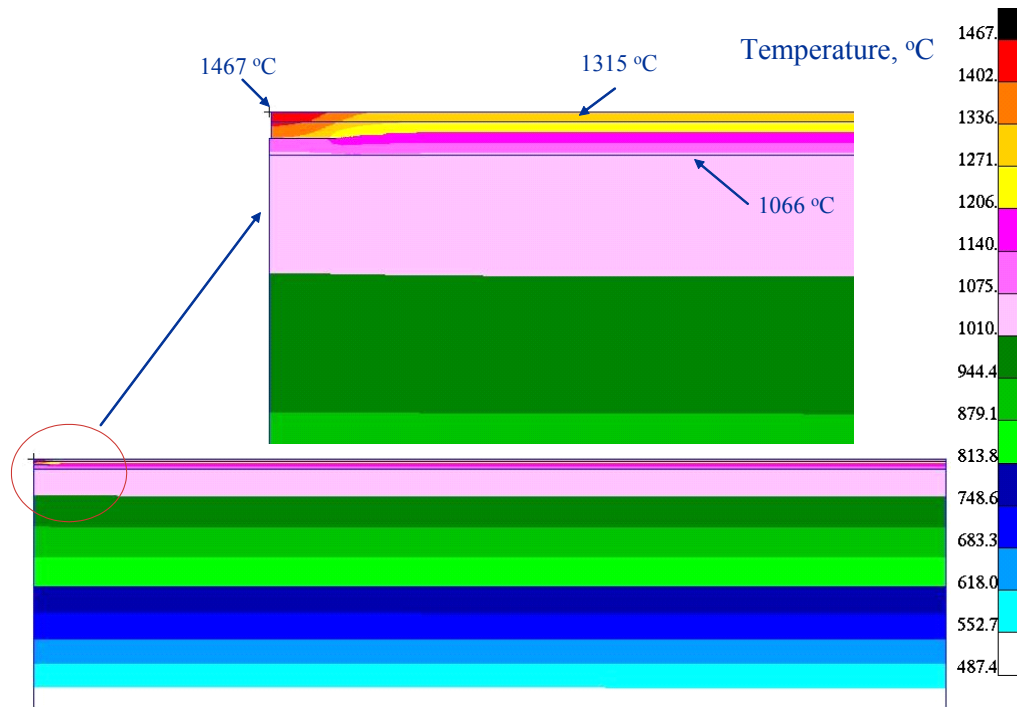


Evans and Hutchinson model,  
Surface Coating  
Technology, 2007

$$G = \frac{1}{6} \left( \frac{1+\nu_1}{1-\nu_1} \right) E_1 h (\alpha_1 (T_s - T_0))^2$$

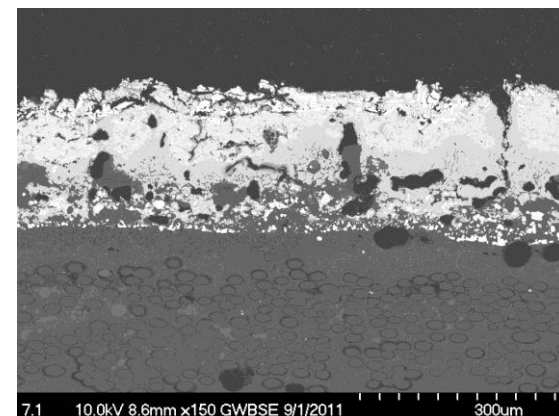
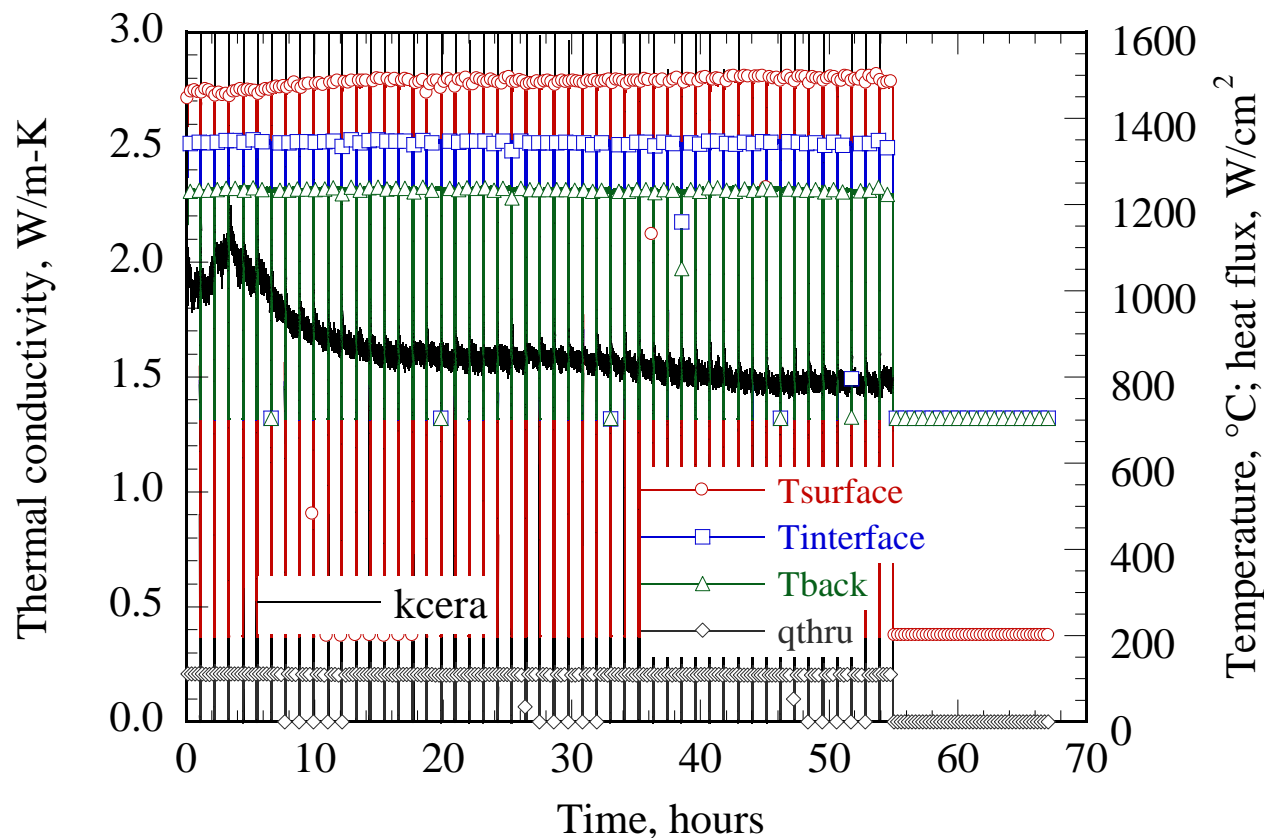


The FEM model



# Thermal Gradient Cyclic Behavior of Air Plasma Sprayed $\text{Yb}_2\text{SiO}_5$ (with $\text{HfO}_2$ Composite)/ $\text{Yb}_2\text{Si}_2\text{O}_7$ /HfO<sub>2</sub>-Si Coatings on SiC/SiC CMCs

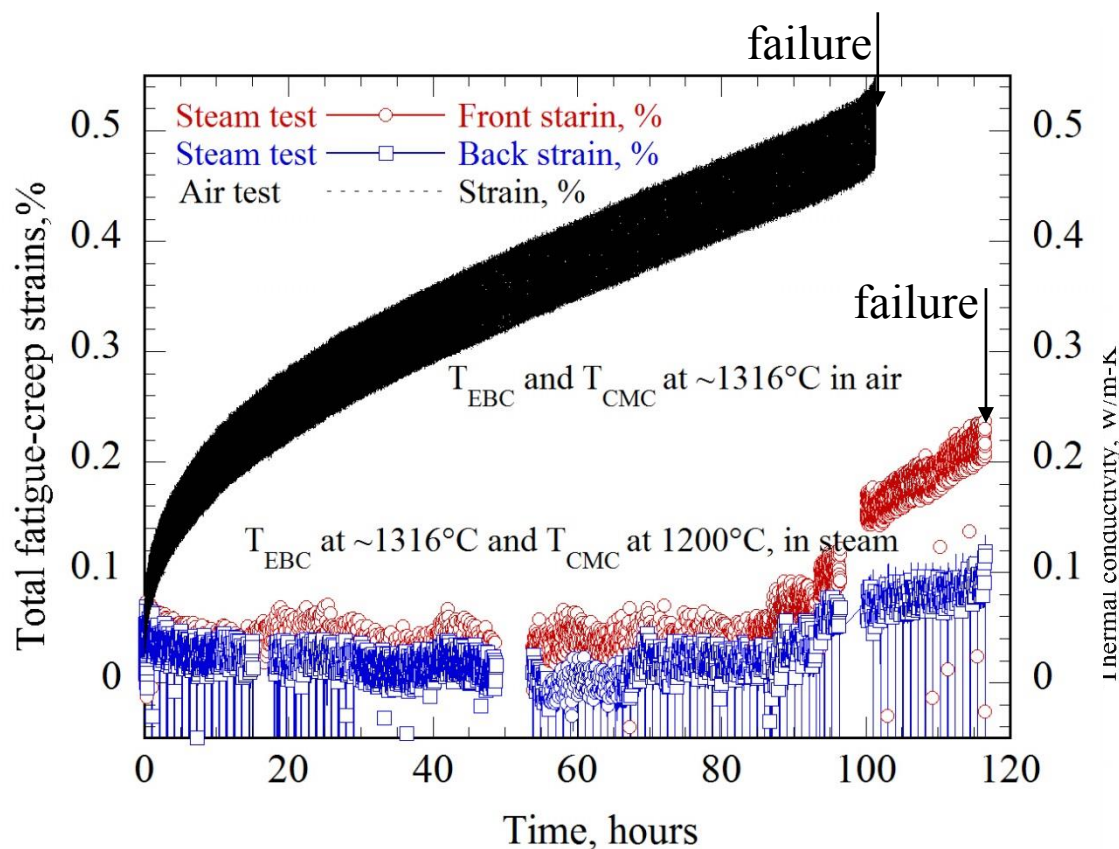
- $T_{\text{surface}} \sim 1482\text{--}1500^\circ\text{C}$ ,  $T_{\text{interface}} 1350^\circ\text{C}$ ,  $T_{\text{back surface}} 1225^\circ\text{C}$ , heat flux  $110\text{ W/cm}^2$
- Localized pore formation



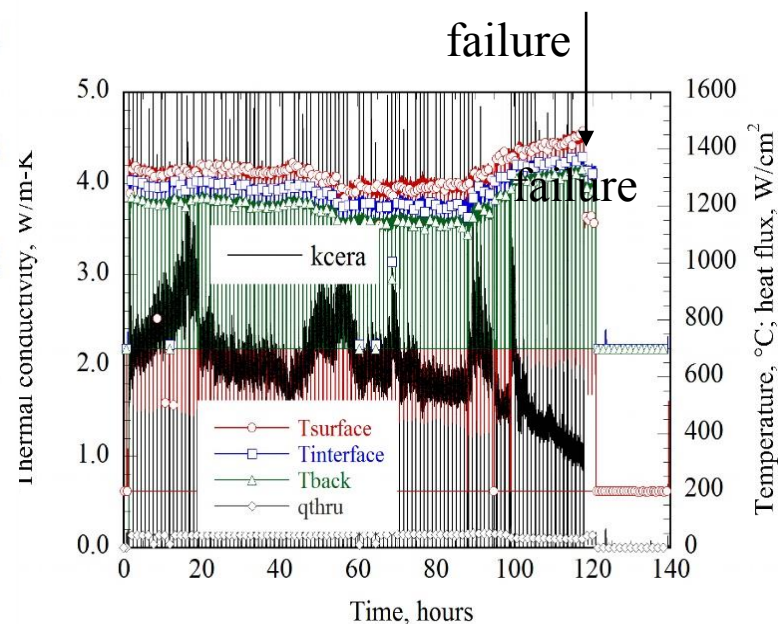
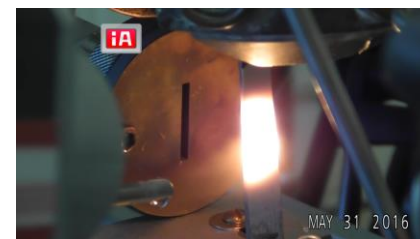
After 50hr Cyclic Testing

## Fatigue Testing using a Laser High-Heat-Flux Approach for Environmental Barrier Coated Prepreg SiC/SiC CMCs

- Environmental Barrier Coatings  $\text{Yb}_2\text{SiO}_5/\text{Yb}_2\text{Si}_2\text{O}_7/\text{Si}$  on MI Prepreg SiC/SiC CMC substrates
- One specimen tested in air, air testing at  $1316^\circ\text{C}$
- One specimen tested in steam, steam testing at  $T_{\text{EBC}} 1316^\circ\text{C}$ ,  $T_{\text{CMC}}$  at  $\sim 1200^\circ\text{C}$
- Lower CMC failure strain observed in steam test environments



Fatigue strains (amplitudes) – Time Plot

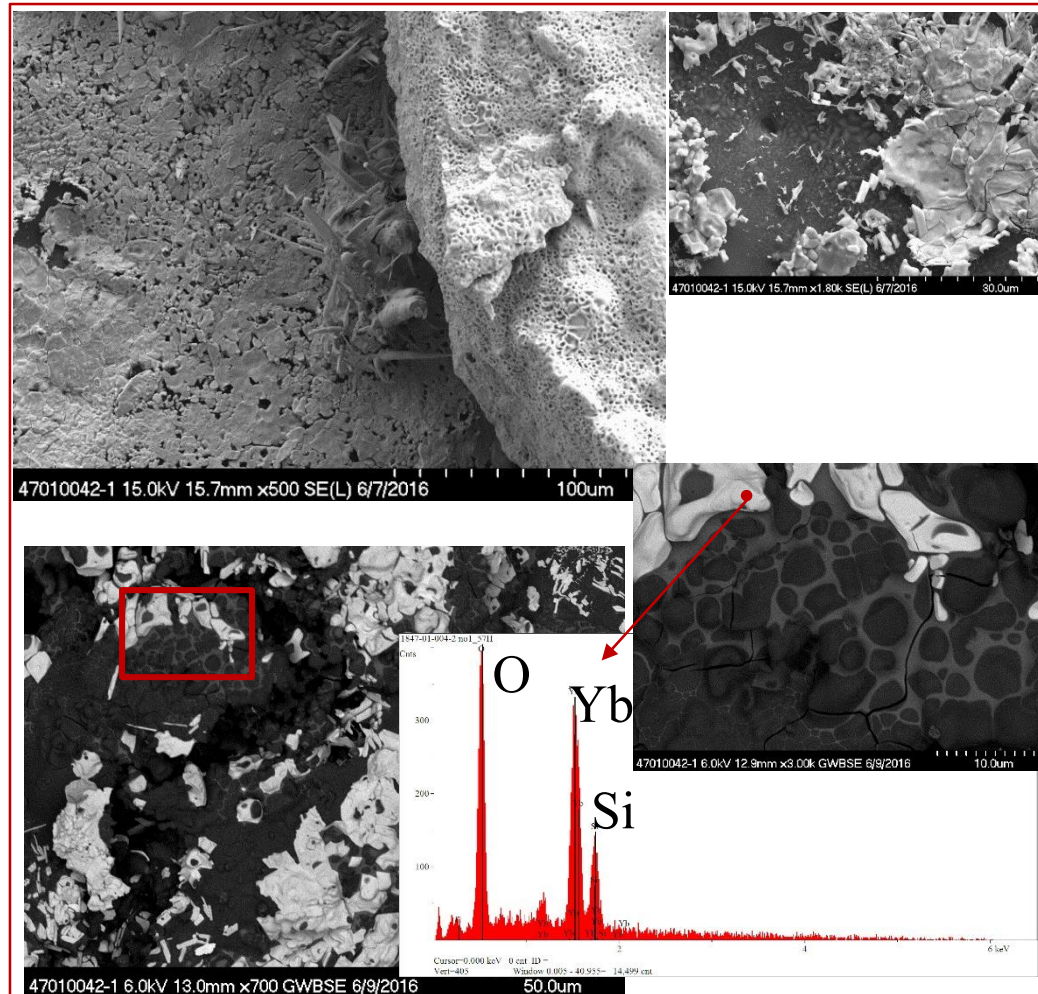
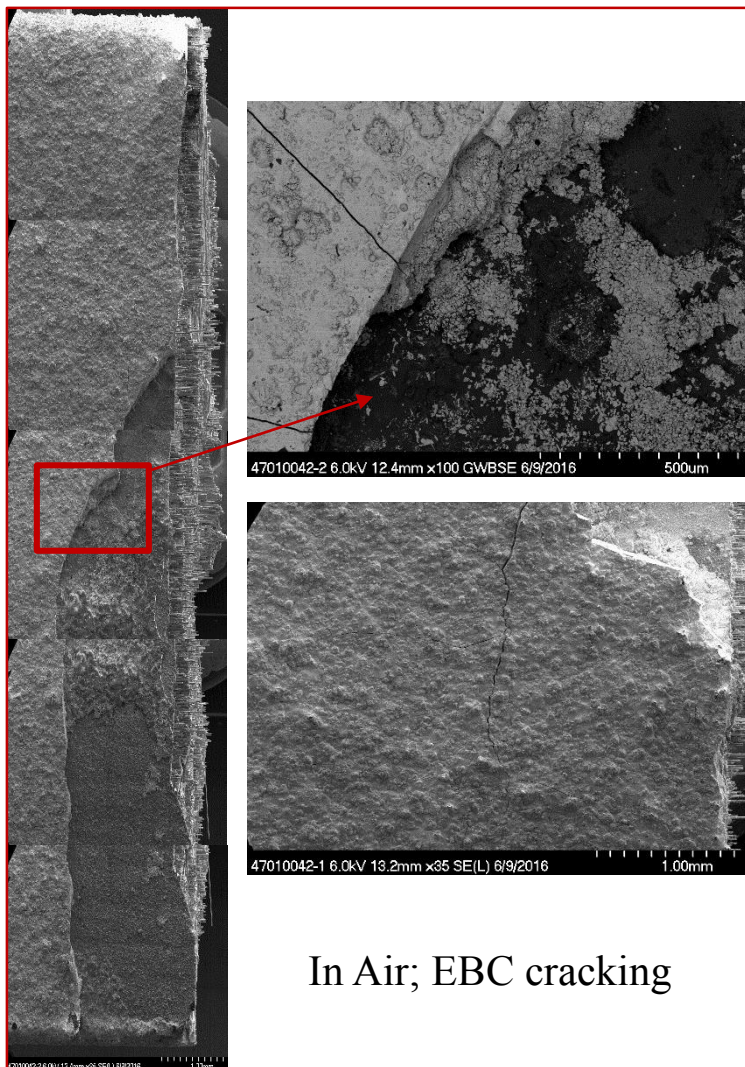


Thermal conductivity – Time Plot



# Fatigue Testing using a Laser High-Heat-Flux Approach for EBC Coated Prepreg SiC/SiC CMCs - Continued

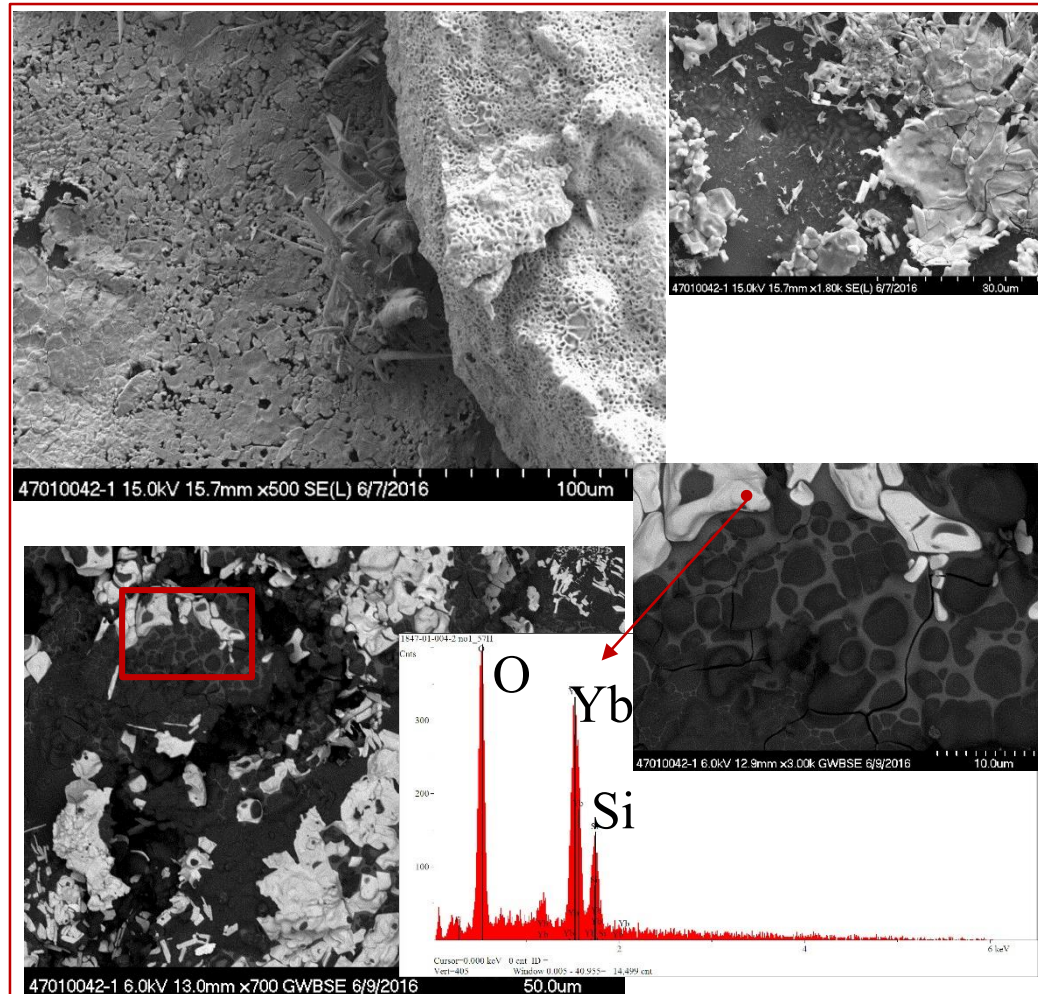
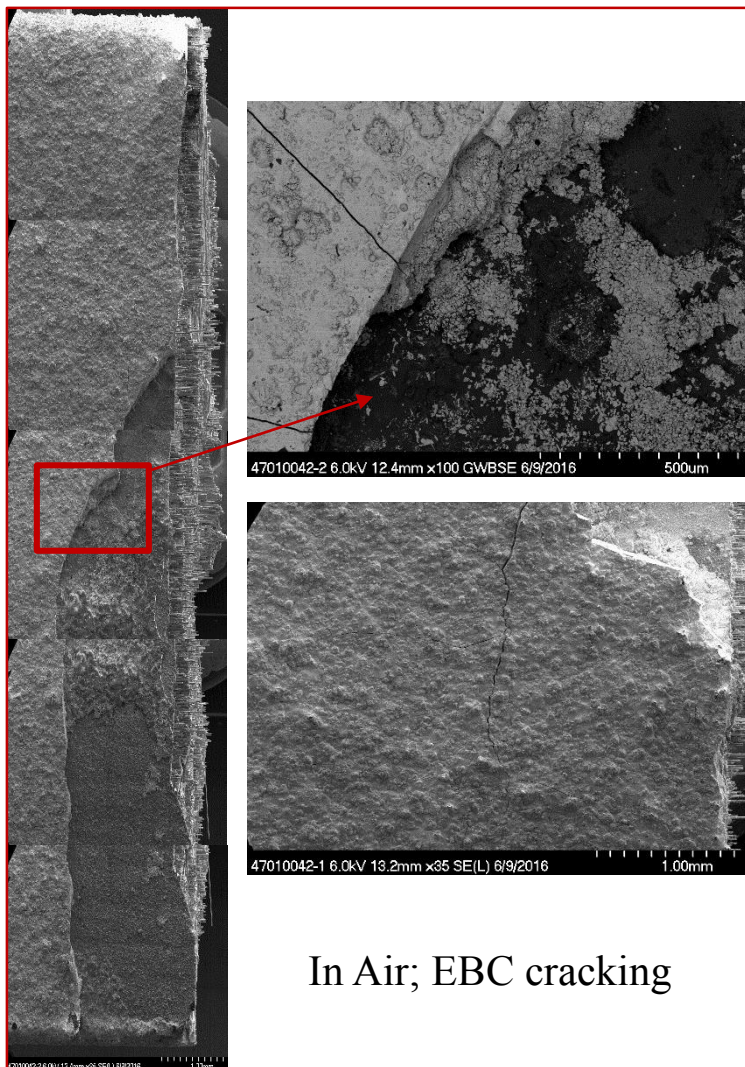
- Crack and recession failure in air and steam tests





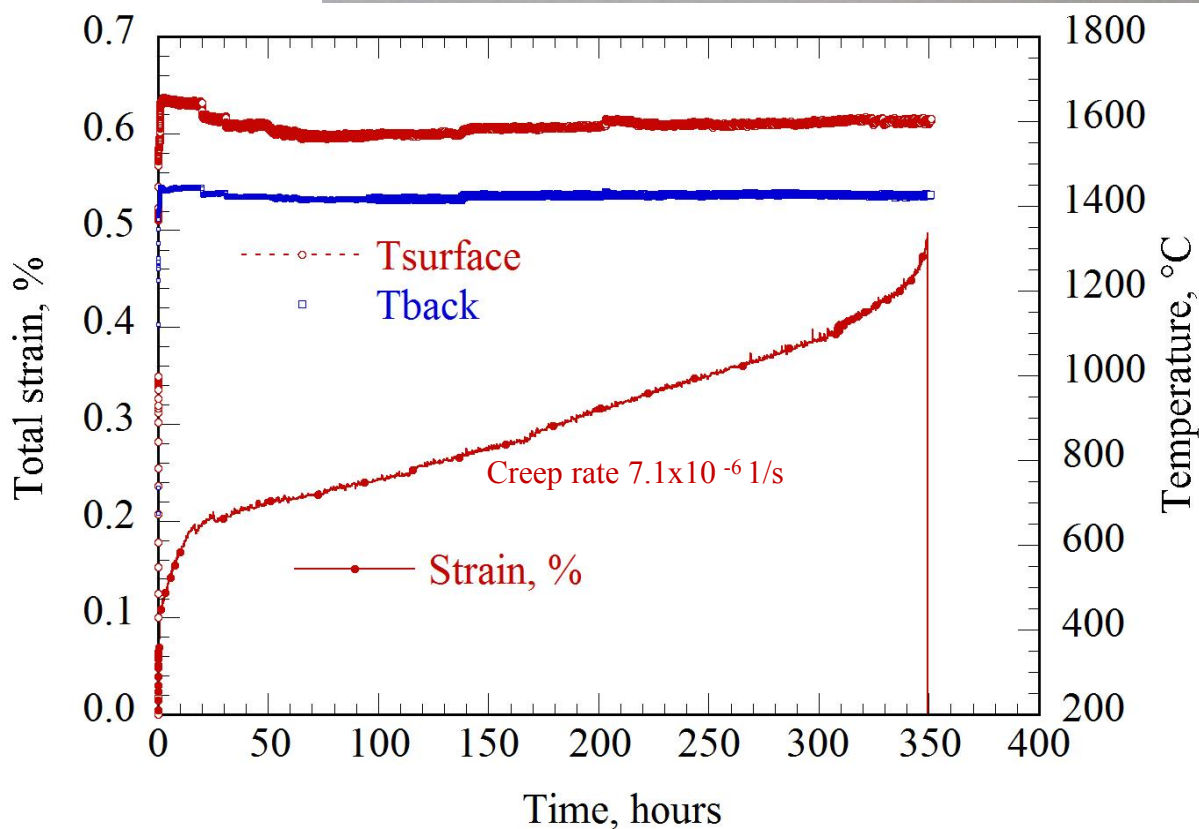
# Fatigue Testing using a Laser High-Heat-Flux Approach for EBC Coated Prepreg SiC/SiC CMCs - Continued

- Crack and recession failure in air and steam tests



# EBC Coated CMC 2650°F (1454°C) Creep Rupture Durability Test

- SiC/SiC CMC 12C-470-022 SiC/SiC CVI-MI CMC specimen
- Coated with 2700°F (1482°C) RESi and Rare Earth EBC
- Test temperatures:  $T_{\text{EBC surface}}$  at 2850-3000°F (1600-1650°C), and  $T_{\text{cmc back}}$  at ~2600°F (1426°C)



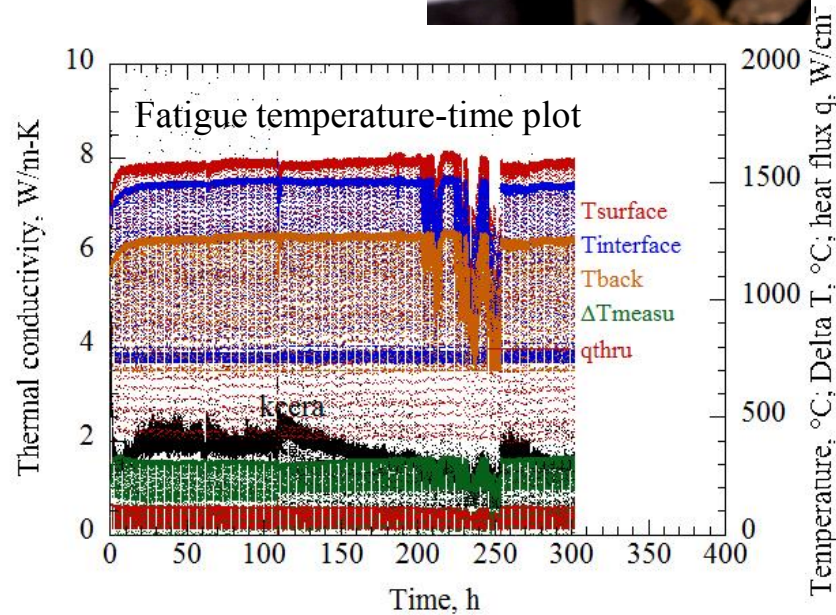
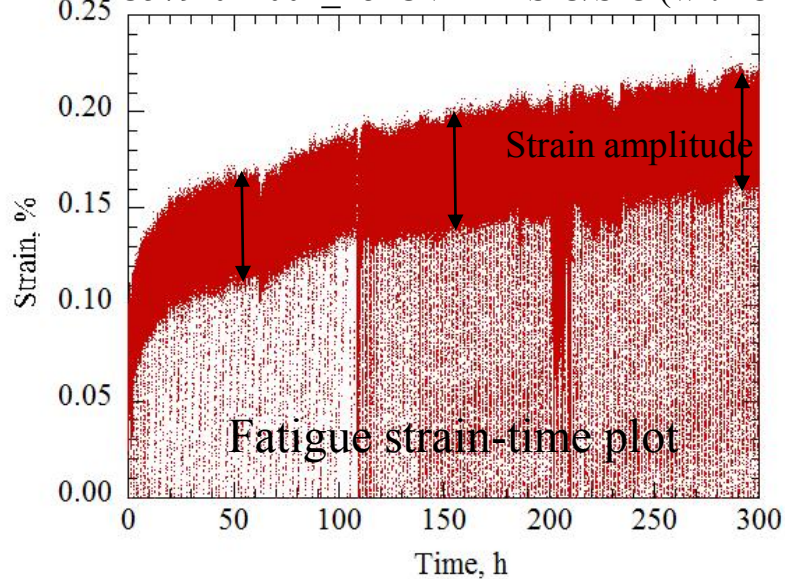


# Advanced EBC-CMC Fatigue Test with CMAS: Successfully Tested 300 h Durability in High Heat Flux Fatigue Test Conditions

- A thin EB-PVD turbine airfoil EBC system with advanced  $\text{HfO}_2$ -rare earth silicate and GdYbSi (controlled oxygen activity) bond coat tested at  $T_{\text{EBC-surface}} 1537^\circ\text{C}$ ,  $T_{\text{bond coat}} 1480^\circ\text{C}$ ,  $T_{\text{back CMC surface}} 1250^\circ\text{C}$
- Fatigue Stress amplitude 69 MPa, at mechanical fatigue frequency  $f=3\text{Hz}$ , stress ratio  $R=0.05$
- Low cycle thermal gradient fatigue 60min hot, 3min cooling

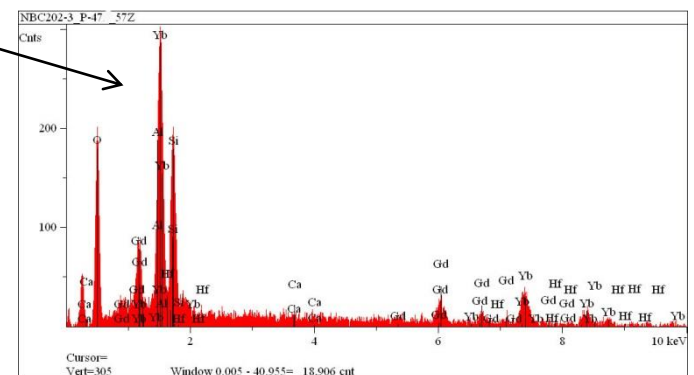
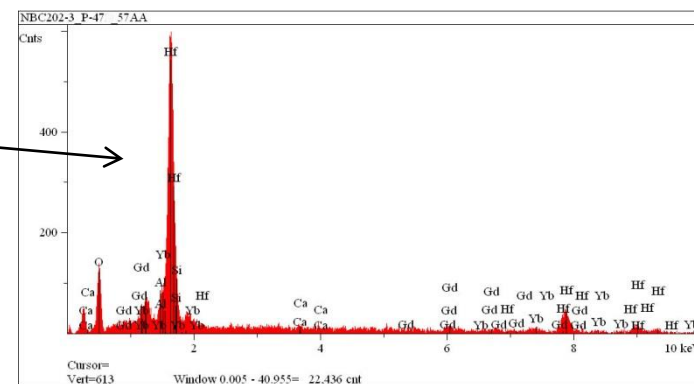


1537°C, 69MPa (10ksi), 300 h fatigue (3 Hz,  $R=0.05$ ) on  
14C579-011001\_#8 CVI-MI SiC/SiC (with CMAS)



## EBC Fatigue Test Failure with CMAS

- Advanced alternating  $\text{HfO}_2$ -RE-silicate coatings (EB-PVD processing) –  $\text{HfO}_2$ -layer infiltration and rare earth silicate layer melting
- Advanced composition clustering EBCs being developed

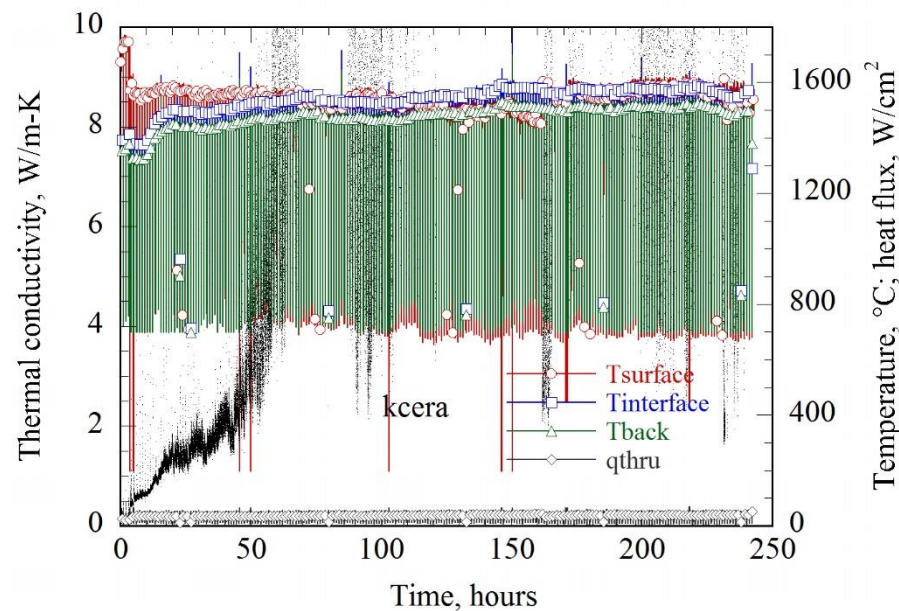
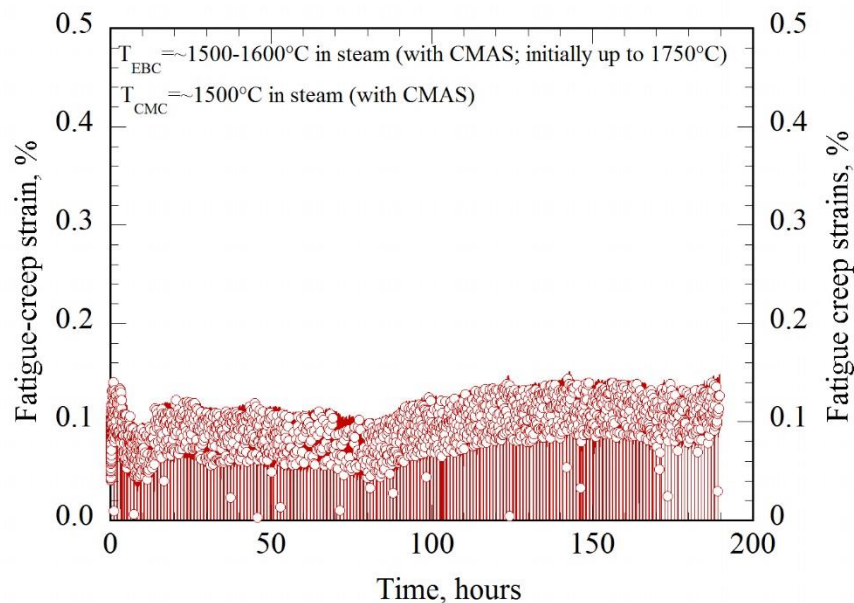
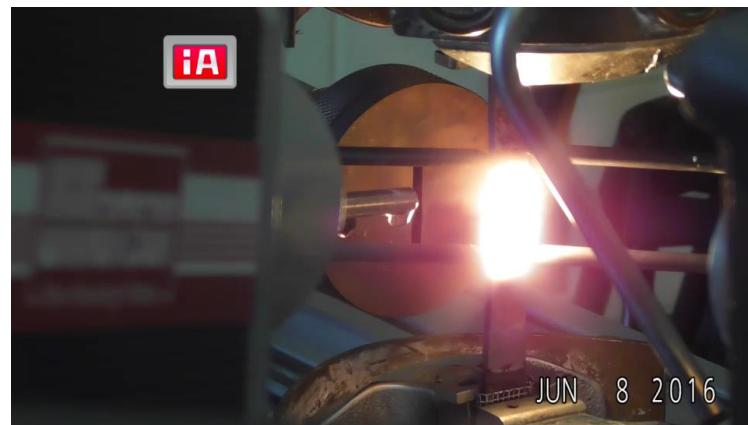
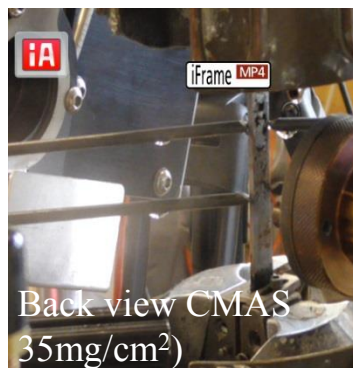
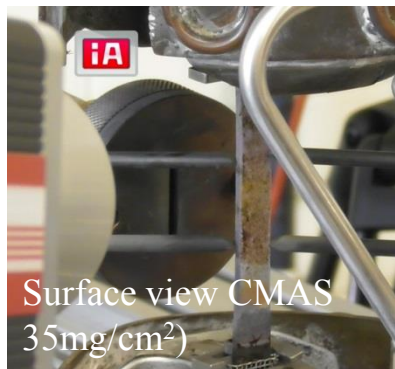


EB-PVD Processed EBCs: alternating  $\text{HfO}_2$ -rich and ytterbium silicate layer systems for CMAS and impact resistance



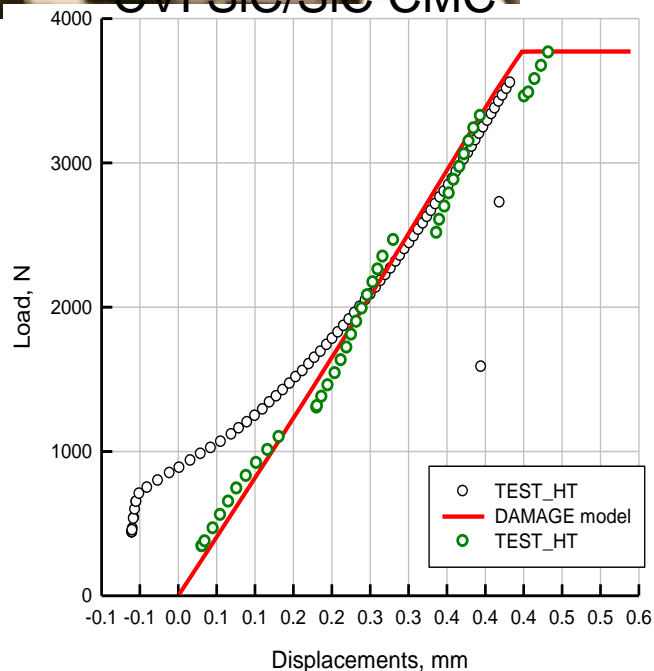
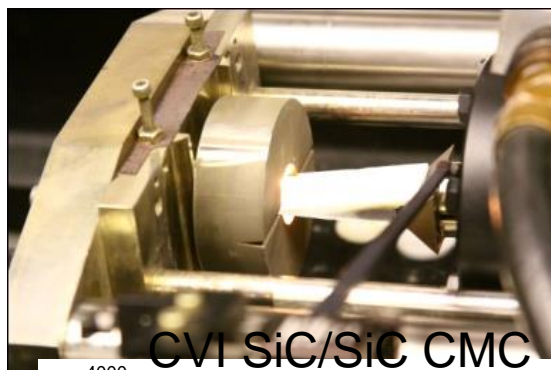
# Advanced EBC-CMC Fatigue Test with CMAS and in steam Jet: Successfully Tested 150 h Durability in High Heat Flux Fatigue Test Conditions

- Advanced Hf-NdYb silicate-NdYbSi bond coat EBC coatings on 3D architecture  
CVI-PIP SiC-SiC CMC (EB-PVD processing)

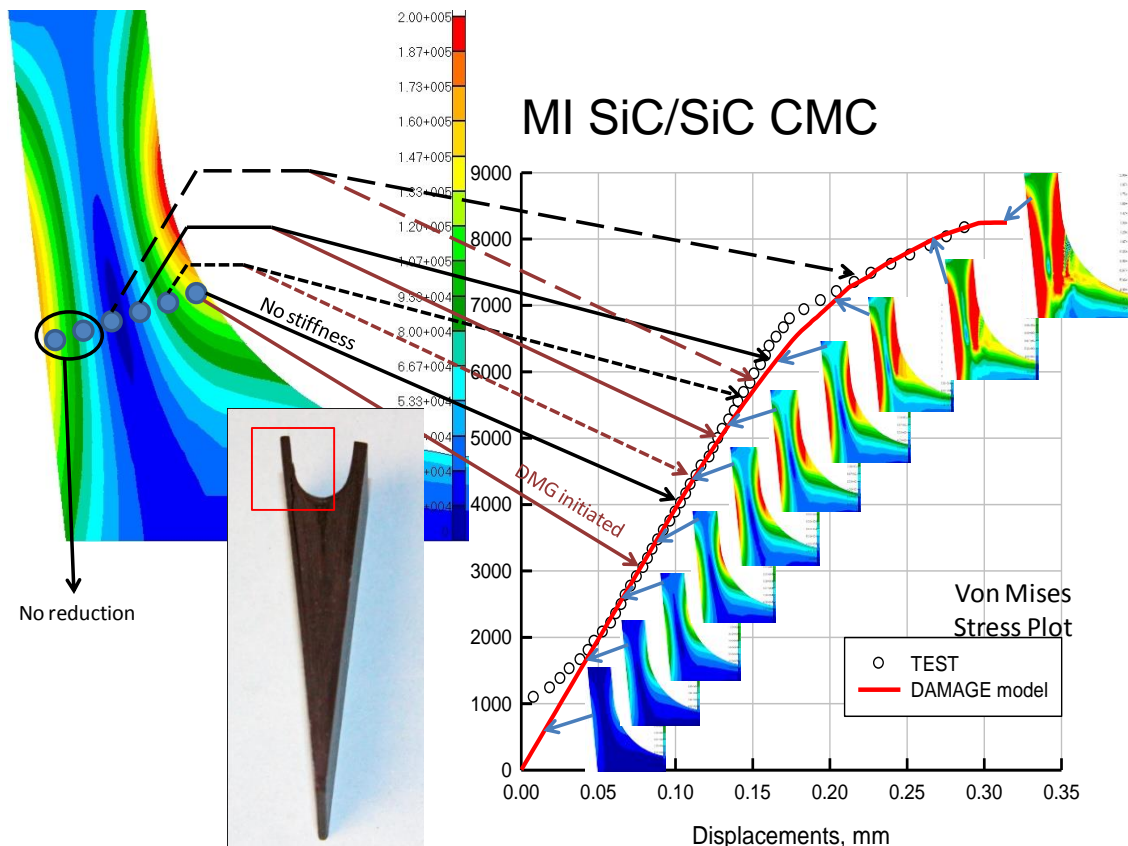


## SiC/SiC Turbine Airfoil Trailing Edge Tests

- Subelement wedge testing and high temperature tests, aiming at understanding the CMC and EBC degradation



Subelement Load-Displacement curve – CVI CMC trailing edge



Subelement Load-Displacement curve – Prepreg MI CMC trailing edge

## Summary and Future Plans

- **Advanced high heat flux creep rupture, fatigue rigs established for simulated turbine engine EBC-CMC testing**
  - High temperature comprehensive environment testing capability including heat flux, steam and CMAS, at very high temperature
  - Real time coating degradation monitoring and fatigue-creep stain monitoring
  - Testing capabilities incorporated into the advanced EBC-CMC developments
- **Long term creep rupture and fatigue behavior evaluated for Hafnium Rare Earth silicate and Rare Earth-Silicon based EBCs-CMCs at 1482°C+ (2700°F+)**
  - Crucial for advanced EBC-CMC development and validations
- **The heat flux thermomechanical testing of subelements for the EBC-CMC subelement**
  - Important for durability and life modeling

### Future plans

- HCF high heat flux rig with additional environmental testing capabilities (steam-air mixture environments and controlled steam or vacuum capabilities)
- EBC erosion-impact capabilities also planned in combination of laser high heat flux, creep-fatigue, high velocity steam, and CMAS integrated tests
- Additional full field strain measurement experiments, in particular at high temperatures
- Planned a multi-axial testing rig for CMC and EBC testing



## Acknowledgements

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Terry McCue, Serene Farmer, Francisco Solá, SEM and TEM

Valerie Wiesner and Narottam Bansal, Gustavo Costa: Fundamental CMAS behavior